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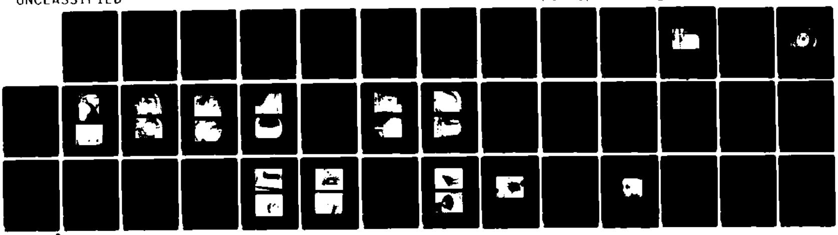

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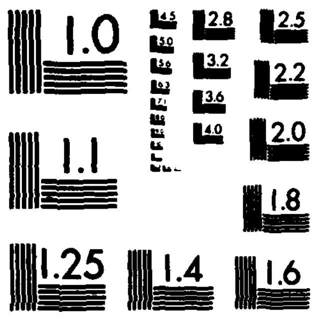

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DEVELOPMENT AND FABRICATION OF COLLAPSIBLE FUEL STORAGE DRUMS FOR ARCTIC USE

N. J. Abbott, R. E. Erlandson
Albany International Research Co., Dedham, MA 02026

Final Report

September 1980 - December 1983

Contract No. DAAK70-80-C-0214

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Materials research and end item development was conducted contractually to produce 500 gallon fuel storage drums capable of satisfying a need for serviceability at -60°F as components of the Arctic Forward Area Refueling Equipment (AFARE) system. A novel drum fabrication technique, combining the concepts of braided Kevlar fabric reinforcement and injection molding of a polyurethane elastomeric coating was employed to successfully produce two prototype drums. Failure of the drums during Arctic field testing was attributed to crystallization of the urethane coating elastomer after prolonged exposure below 0°F.

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Preface

This report describes work performed and results obtained under Contract DAAK70-80-C-0214, for the Belvoir Research and Development Center, Fort Belvoir, VA, 22060. Mr. Paul E. Gatza, of the Rubber and Coated Fabrics Research Group, Materials, Fuels and Lubricants Laboratory, was the Contracting Officer's Representative. Appreciation is extended to him and other Belvoir Research and Development Center personnel, who provided technical guidance and performed certain screening and verification testing relative to selection of materials and drum fabrication procedures.

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DEVELOPMENT AND FABRICATION OF COLLAPSIBLE FUEL STORAGE DRUMS FOR ARCTIC USE

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Introduction

As part of their Arctic Forward Area Refueling Equipment System, the U. S. Army uses a 500-gallon capacity collapsible fuel drum which can be dropped into remote areas from a helicopter and then rolled over the terrain for short distances to a temporary storage depot. The drums presently available use an elastomer system which becomes stiff and brittle at extreme Arctic temperatures (-60°F). The first objective of the work was to develop an elastomeric coating compound which would remain functional at these low temperatures, as well as having the fuel resistance and durability characteristics required in this application. Fuel drums are currently being made using a modification of tire-building technology. The second objective of the program was to explore the feasibility of employing braiding and elastomer casting technology to produce a drum which meets the requirements outlined in MIL-D-23119D.

Phase I: Development of Elastomeric Coating and Braided Reinforcing Structure

This portion of the report describes the first phase of this work, which was concerned with development of a suitable elastomeric coating system, and a braided reinforcing structure having the desired characteristics.

Braiding Technology

The manufacture of a drum according to the requirements of MIL-D-23119D provides an ideal opportunity to utilize in an imaginative way the braiding technology that has been developed within the organization over the past few years. The reinforced elastomer component of the drum is a symmetrical cylindrical shell of length 58" and diameter 55" with partially open ends. The hoop stresses that the reinforcing material must bear in the loaded state are given by the standard expression for pressure vessels, but the longitudinal loads are borne primarily by separate, internal, axially oriented tension members. Accordingly the textile reinforcing fabric can be highly anisotropic, and this particular configuration lends itself to the production by braiding of a unitary, essentially seamless structure.

The use of braids to reinforce long, circularly symmetrical cylinders is well known, and the application of braiding to reinforce large, discrete shapes has been demonstrated very successfully for certain rigid composite structures [1,2,3,4] and good mechanical performance and low production costs have been demonstrated. In order to understand the application of braiding to this application, a brief discussion of the general field of braiding technology is in order, and is presented below.

The braiding process is in essence extremely simple. In its fundamental form, two sets of yarn package carriers are made to traverse undulating circular paths in opposite directions, and the timing of the crossovers is such that each set of yarn interlaces with the other set to form a continuous tubular fabric. The detailed structure of the fabric can be changed by manipulation of the intersection sequence of the loaded carriers just as the structure of a woven fabric can be modified by altering the shedding sequence of the warp yarns. In fact, it is not possible to distinguish between isolated sections of braided and woven fabrics of similar construction. The principal functional difference between a braid and a woven structure (both here assumed to be orthogonal) is that the unit cell of the structure is oriented at 45 degrees to the direction of fabric production in the braid, and is parallel to the direction of fabric production in the woven fabric. The braided structure is completely radially symmetrical, and because of its orientation is highly deformable in shear by forces that are parallel to the axis of production. It is this feature that makes braiding an ideal process for the reinforcement of flexible hoses and cables.

The basic braiding process described above is capable of several modifications that enormously increase the versatility of the technique. As customarily carried out, the process is limited to the reinforcement of cylindrical hoses of uniform diameter, and the number and sizes of the component yarns are chosen to give a good coverage of the reinforced elastomer, together with an adequate load bearing capability. However, it is possible to braid over a rigid mandrel of varying cross-sectional shape and to obtain an automatic conformal fitting of the braid fabric to the discrete mandrel shape. Moreover, by the use of a formation ring and reversing drive in conjunction

---

[1] Sanders, L. R., McDonnell Aircraft Company, "Braiding - A Mechanical Means of Composite Fabrication," MCAIR No. 76-019, presented at the Eighth National SAMPE Conference, 12-14 October 1976.

[2] Post, R. J., McDonnell Douglas Astronautics Company, "Braiding Composites - Adapting the Process for the Mass Production of Aerospace Components."

[3] Sanders, L. R., McDonnell Aircraft Company, "Manufacturing Methods for Low Cost Automated Fabrication of Composite Structures," IR-421-5(VII), Seventh Quarterly Interim Technical Report, January 1977.

[4] Stifel, P. M., McDonnell Aircraft Company, "Manufacturing Methods for Low Cost Automated Fabrication of Composite Structures," IR-421-5(VIII), Eighth Quarterly Interim Technical Report, April 1977.

with a mandrel, it is possible to traverse the workpiece through the machine past the fabric formation point in both directions sequentially, so as to lay up on the mandrel a series of layers of fabric in which the yarns are continuous throughout, and there are no major discontinuities at the turnaround points. This has obvious advantages in connection with the production of short braided structures intended for pressure vessel applications.

Another modification that can be of value in the production of pressure vessels is the incorporation of additional axial yarns into the braided structure. These yarns are supplied from a set of stationary package holders mounted parallel to and slightly below the rotating set of holders, and the fabric that is produced is triaxial in nature - that is, it consists of three distinct sets of threads as opposed to the customary two sets in conventional woven fabrics. This feature gives enhanced design capabilities: for example, if the same yarn is used in all three directions and the sets are arranged to be oriented 60 degrees apart, a structure is produced that is very close to isotropic in its mechanical properties; alternatively, if there is only a small angle between the axial yarns and the moving carrier yarns then the structure approximates to a uniaxial tape; and as another limiting alternative configuration, the moving carrier yarns can be aligned at a high angle to the axial yarns, thus producing a structure that is quite similar to a conventional woven fabric. It is this latter type of structure configuration that is of interest in the present application.

In the braiding process as it is normally carried out as a textile process the carriers revolve in a horizontal plane, and the direction of fabric production is vertical. This is a perfectly satisfactory configuration for the production of flexible materials that will be subsequently wound on a take-up package, but it is very inconvenient for braiding over a mandrel for the production of rigid components. In order to simplify this process the braider can be tipped through 90 degrees so that the plane of rotation of the carriers is vertical and the long axis of the mandrel is horizontal. This maneuver also facilitates the design of mandrel holding and traversing mechanisms, and minimizes the limitations on the length of the article that can be produced. The maximum diameter of the component that can be accommodated in the braider is set by the geometry of the carrier bed and yarn guides. Our current arrangement enables us to braid over mandrels that are up to ten feet long and sixty inches in diameter, so the fuel drum falls well within the geometrical capability of the technique. A good illustration of this type of braiding is the picture of our braider shown in Figure 1.

The extent of coverage that can be achieved on a single pass through the braider is determined by many interrelated factors: the diameter of the workpiece, the rate of traverse, and hence the helix angle of the moving carrier yarns, and the width of the individual yarns as they lie in the workpiece subsequent to the fabric formation. This latter parameter is controlled by both the linear density of the yarn and the amount of twist in the yarn: a low twist yarn is able to flatten out to give a cross-sectional aspect ratio of up to 10:1, which ensures high transverse coverage and low thickness; a yarn with twist is not able to accept this type of cross-sectional distortion and a lower cover, thicker fabric results. In composite applications the flatter yarn sections are usually more desirable since the uniformity of the product is improved and the number of voids in the structure is minimized.

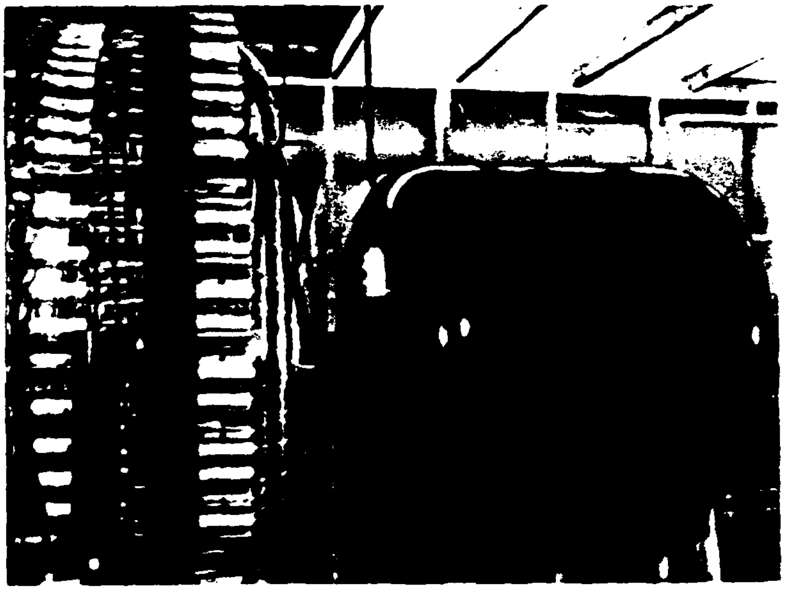


Figure 1. Coated Mandrel Mounted in Braider

The maximum yarn linear density that can be accommodated in our present braider is set by the size of the carrier guides and fair-leads, which limit the maximum width of the flattened strands to approximately 0.2 inch. It is not normally possible to use very high helix angles since this leads to fabrics with highly anisotropic load bearing capability. In the present application, however, such anisotropy is not deleterious, and can, in fact, be exploited to advantage.

Proposed Drum Fabrication

The general procedure for manufacturing the drum consists of spray coating the inner liner onto a mandrel conforming to the size and shape of a filled drum, building the fiber reinforcement onto the coated mandrel, enclosing the whole assembly in a mold, and casting the cover using standard mold casting techniques. Finally, the mandrel will be broken down and removed through the two end holes in the drum.

Actual drum fabrication will not be carried out until Phase II of the work. The brief description above has been included here in order to clarify some of the requirements which must be met by the elastomeric coating compound as well as the fiber reinforcement. Specifically, the coating must be suitable for casting as well as for spraying in the form of a solution. The fiber reinforcement must meet the mechanical requirements of the specification, as well as being open enough to permit good penetration and impregnation in the casting process.

Braid Development

In order to facilitate the development of a reinforcing structure which will provide the desired mechanical characteristics, a simple test mandrel was constructed from wood and sheet aluminum. This mandrel was octagonal in shape, and its perimeter was equal to the circumference of the 55 inch diameter fuel drum. The radii at the ends of the mandrel, as can be seen in Figure 2, were approximately the same as those of the fuel drum. To conserve material, the mandrel length is only about two-thirds that of the fuel drum. The primary function of this mandrel was to make possible preparation of drum material samples that were essentially the same as those proposed for ultimate fabrication over a cylindrical mandrel, with an important and desired exception being that they were planar because of the octagonal, rather than circular, cross-sections of the mandrel. These samples greatly facilitated physical testing of the braid/woven fabric/elastomer laminate that was produced from them. The approximations made in the size and shape of this first mandrel, in comparison with those of the fuel drum, ensured that structural details such as braid angles need not be changed significantly later. Another advantage is the fact that much of the hands-on experience gained during fabrication of the test samples was directly applicable to fuel drum fabrication.

The mandrel was supported horizontally on a 3" diameter steel tube held at each end on a traversing carriage. It was critical that the mandrel be accurately centered in the braider ring, and that deflection, as well as any movement during braiding, be kept to a minimum. This was accomplished by using a rigid support, and adjusting and locking the mandrel in the proper

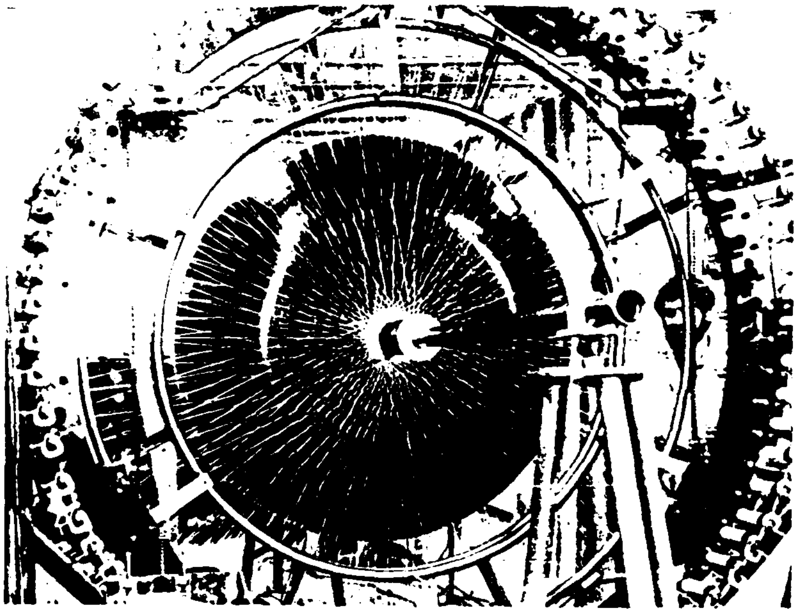


Figure 2. Braid Formed for First End of Drum

position on this support. A motor-driven carriage traverse permitted the mandrel to be passed back and forth through the braider ring. It was necessary also to provide for slow braider operation in order that the speed with which yarn was withdrawn from the braider packages did not exceed that which was consistent with good tension control. Guide rings also had to be made and mounted to ensure that the braid angle remained constant throughout the while of a forward-reverse traverse cycle.

One problem which had to be faced immediately was the prevention of yarn slippage on the shoulders of the mandrel, particularly at the point on the shoulder where the braid angle changes abruptly, at the transition from the body to the end of the drum. This was eventually solved by using a 100% solids adhesive based on Adiprene 167 and Caytur 21 which could be painted on and cured with a heat gun to the tacky state. It was important, of course, that the adhesive be fully compatible with the elastomeric coating to be used. Since this was to be a urethane, the use of a urethane adhesive ensured excellent bonding between coating and adhesive.

The first formulation tried was Adhesive Formulation 2642-21-1:

Part A - Adiprene L167	100
Part B - Caytur 21	31.7

Preheat part A to 160°F, add Part B and de-gas. Activate adhesive by heating to 350°F with heat gun.

This formulation was too brittle at -60°F and formulation 2642-21-2 was tried:

Part A - Adiprene L100	100
Benzoflex 9-88SG	10
Multiflow	0.1
Part B - Caytur 21	19.6

Formulation 21-2 was still flexible at -60°F and worked well as the braiding adhesive.

The braided reinforcement was made from 1500 denier Kevlar 29 yarn, and in order to provide axial direction support 7000 denier Kevlar yarn was laid in at each of 144 positions around the circumference of the drum. This forms a triaxial structure as shown in Figure 3, which represents what we decided would provide the best opportunity to build a final structure having the desired properties by simply adding a suitable number of braided reinforcement layers by traversing back and forth an appropriate number of times. Each traverse will increase the cover provided by the structure because no two layers would be expected to lie in perfect registration with each other. Figure 4 shows, for example, the increase in cover resulting from two layers of braid (compare with the single braided layer in Figure 3). Figures 5 to 10 show the mandrel mounted in the braider during the braiding operation, and illustrate the manner in which the braided structure is brought over the end of the drum and around the supporting shaft to provide reinforcement for each end of the drum. Traverse reversal can thus be accomplished without any break in the reinforcing yarns.

Figure 3. Applying Urethane Adhesive at Shoulder

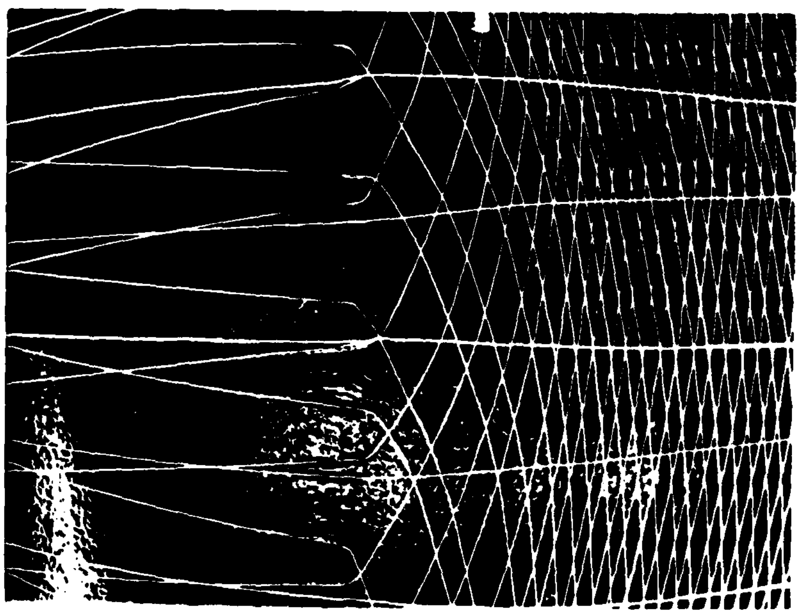


Figure 4. Close-up of Shoulder, Showing Abrupt Change in Yarn Direction

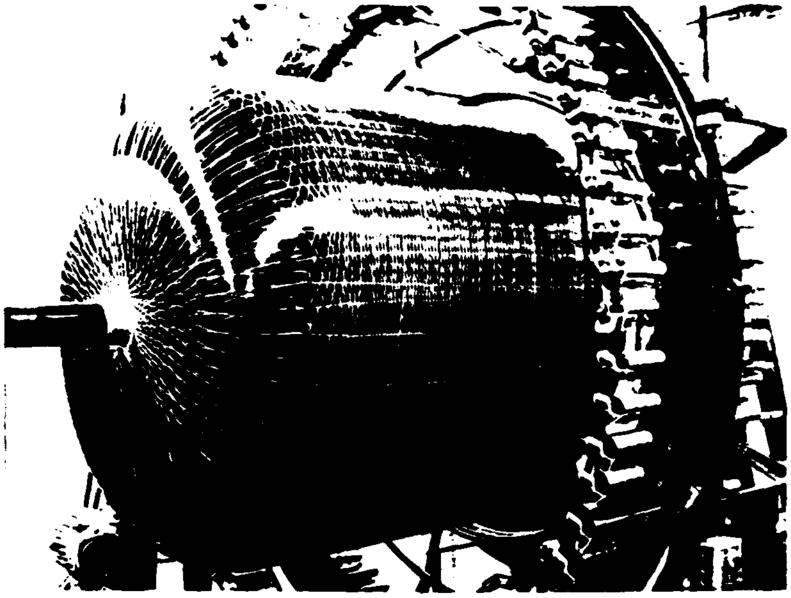


Figure 5. Completed First Layer of Braid over Drum Body

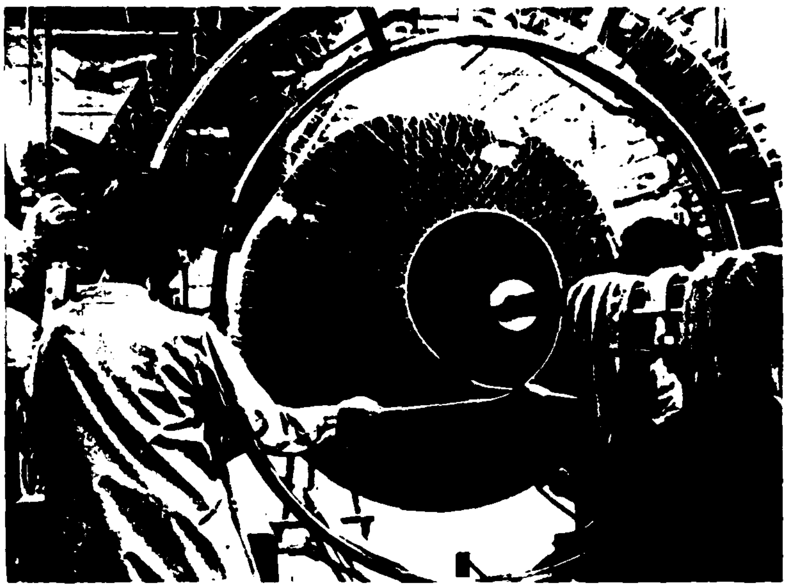


Figure 6. Closing First Layer of Braid over End of Mandrel

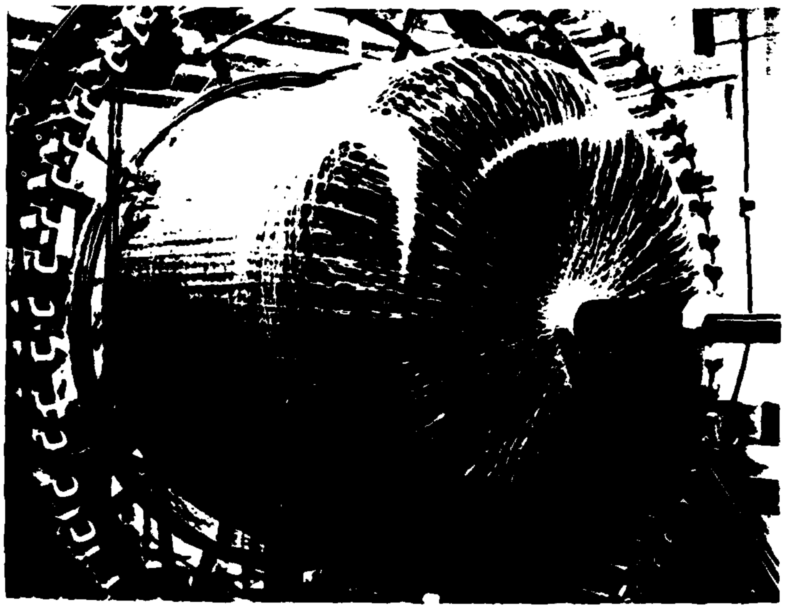
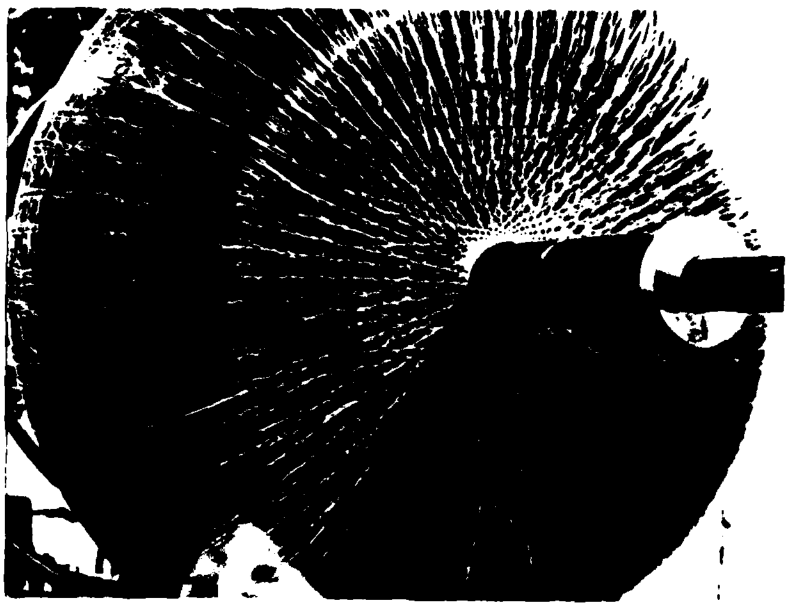


Figure 7. Applying 4th Layer of Braid



Figur ers of Braid - Drum End

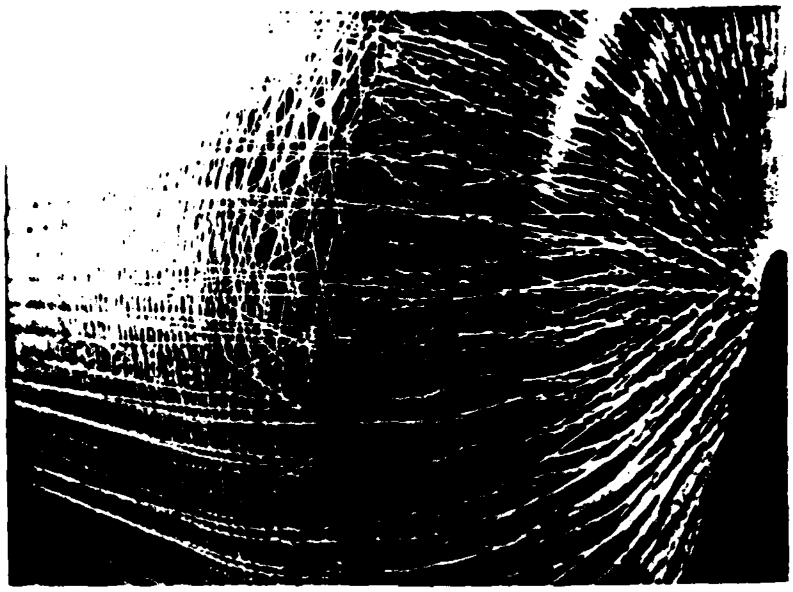


Figure 9. 4 Layers of Braid - Drum Shoulder

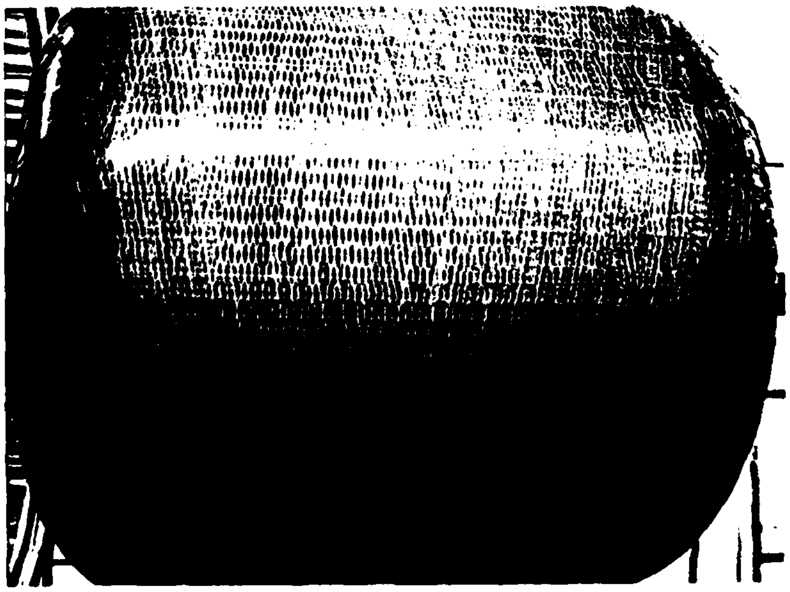


Figure 10. 4 Layers of Braid - Drum Body

It was realized, however, that this relatively open structure which is ideal for good penetration of the coating may not be sufficiently continuous to ensure uniform puncture resistance over the whole surface of the drum. Improved puncture resistance was obtained by making a large, tubular knitted fabric from 400 denier Kevlar yarn which could be pulled over the entire mandrel and gathered down at each end to be attached around each end hole. The fabric was made on an 81" wide, 12 gauge Universal flat bed knitting machine and proved to be easy to stretch over the drum after several layers of braid had been added. It was then possible to continue adding layers of braid on top of the knitted reinforcement to build up the desired total tensile strength.

Initial trials of this composite structure are illustrated in Figures 11, 12, and 13. Figure 11 shows two layers of braid made in the usual way. Figure 12 shows a section of the knitted fabric reinforcement. In Figure 14, a test panel has been assembled by laying down 2 layers of braid, covering the top half of the knitted fabric, and laying another two layers of braid over the whole panel. Encapsulation of this panel then provided tests of a 4-layer braid structure with and without the knitted fabric reinforcement. Another panel was also hand laid to give 4 layers of braid, a layer of knitted fabric, another 4 layers of braid, another layer of knitted fabric, and a final 4 layers of braid, to give a total of 12 layers of braid and 2 layers of knitted fabric.

The compound used to encapsulate these panels was the best urethane candidate at that time, described in the coating technology section of this report as formulation 2642-16-1.

The test data in Table 1 were obtained from these samples. They can be compared to tests made on a used drum section made by current tire-building techiques from nylon and neoprene.

Table 1: Characteristics of Braided Test Panels

	Sample 1	Sample 2	Sample 3	Sample 4
Construction	4 braid	4 braid/ 1 knit	12 braid/ 2 knit	4 plies tire cord
Tensile Strength (lb/in) Circumferential Direction	545	680	1230*	950
Puncture Resistance**(lb)	163	187	238	272

*Value lower than actual due to tension unbalance in hand lay-up.
**per MIL-T-6396C, paragraph 4.6.17.

A calculation of required hoop strength based on the specified test pressure of 45 psi gave a value of 1200 lb/in. Although this was higher than the value given by the drum carcass which is now being used by the Army. It was decided to fabricate additional test panels on the prototype drum mandrel itself using 12 layers of braid and 2 layers of knitted fabric in order to ensure that the strength exceeded the estimated 1200 lb minimum.

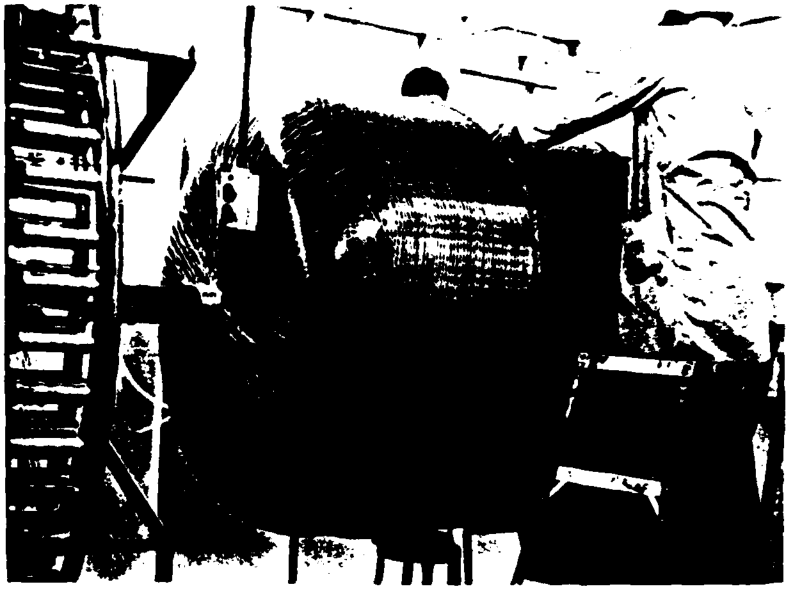


Figure 11. Drawing Knitted Sleeve over Mandrel

Figure 12. Knitted Sleeve Drawn Tight over One End of Drum

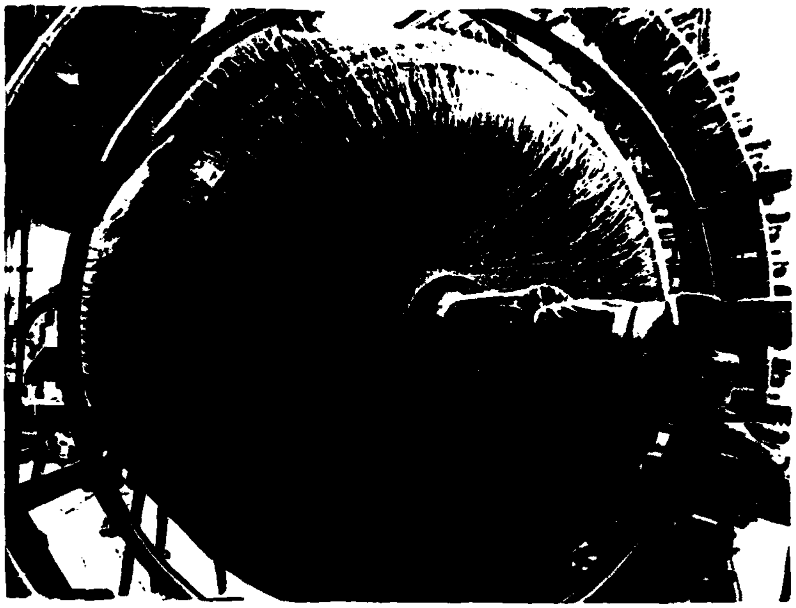


Figure 13. Starting Layer 6 of Braid

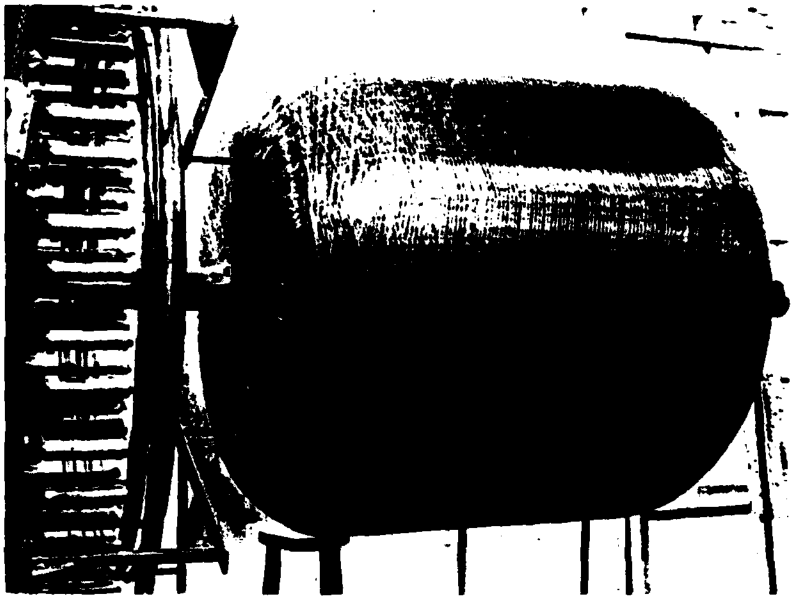


Figure 14. Completed Reinforcement - 8 Layers of Braid

Rectangular 21" x 19" open aluminium frames were taped to the flat faces of the mandrel, as well as being shaped to conform to the shoulder and end sections. After the desired number of layers of braid and knitted fabric had been formed over the mandrel, a urethane adhesive was painted around the circumference of each rectangular frame to impregnate the structure and attach it to the frame. This ensured that tension and geometry were maintained when the frames were cut off the mandrel for impregnation.

These test panels were coated by a casting operation exactly similar to that which will be used for the completed drum. Aluminum plates were clamped to each side of the frame and a two-component urethane system was pumped in using standard liquid injection molding techniques. The resulting impregnated panels were clear and bubble free, and provided good test specimens.

The values in Table 2 were obtained from these specimens:

Table 2: Tests of Molded Panels

		Target Values
Tensile Strength (lbs/in)		
circumferential	1860	1200
axial	1375	600
Puncture Resistance (lb)		
body	406	200
end	354	200

These values are all significantly higher than those obtained from a hand laid-up specimen of similar construction. This is because of better control of tensions, which would be expected to give a more efficient structure. Since the values are all 1-1/2 to 2 times the target value, it is clear that the number of reinforcing layers can be reduced. This will also help to increase the flexibility, which is important because this sample was stiffer than desired, presumably because of the high modulus of Kevlar and the thickness of the reinforcement, which was about 1/4" at its thickest. Impregnation with even a relatively low modulus resin stiffened this structure to what might be an unacceptable level. This would be improved by reducing the thickness of the reinforcement, as well as the amount of fiber which it contains.

An additional trial was made in which the structure was reduced to 4 layers of braid, a layer of knitted fabric, and an additional 4 layers of braid. In order to reduce the axial direction strength, the longitudinal laid-in yarns were reduced to 3 ply 1500 denier Kevlar in place of the 7000 denier yarn. Two test panels were produced in this configuration, and an additional two using 4 layers of braid, one layer of knit, and an additional 6 layers of braid.

We also made a structure of corresponding strength out of high tenacity nylon yarn, to determine the extent to which reducing the yarn modulus (by a factor of 10) would improve the stiffness of the impregnated sructure. This

was made from 1640 denier nylon tire cord in a braid geometry exactly like that used for the Kevlar structure. Sixteen layers of braid were used instead of the 8 used with Kevlar in order to compensate for the reduced strength of nylon in relation to Kevlar. As before, one layer of Kevlar knitted fabric was used in the middle of the structure to improve puncture resistance. Two such panels were made.

One panel of each of these three structure was encased in urethane in the Middletown casting facility. These castings were done using the final version of the urethane compound based on Adiprene M-483.

Test results on these panels are given in Table 3.

Table 3: Test Results on Experimental Panels

Reinforcement	Tensile Strength (lb/in)		Puncture Resistance (lb)
8 layers Kevlar braid	hoop	1140	158
1 layer Kevlar knit	axial	725	
10 layers Kevlar braid	hoop	1620	196
1 layer Kevlar knit	axial	--	
16 layers nylon braid	hoop	1285	210
1 layer Kevlar knit	axial	920	

The strength and puncture values for the nylon sample were adequate. However, rather than being more flexible than the Kevlar samples, it was stiffer. This is because the need for 16 layers of braid, in order to achieve the needed strength, created a structure which was more than twice as thick as the 8 layer Kevlar structure. The relative thinness of the Kevlar structures reduced the bending strains sufficiently to more than offset the effects of Kevlar's high modulus. Moreover, the nylon structure required twice the braiding time of the 8 layer Kevlar, the cost of which more than offset the lower cost of the nylon yarn. Consequently, no more attention was paid to the use of nylon in the drum. It would increase the cost significantly, and would result in a more rigid structure.

The 10 layer Kevlar braid structure had a higher strength than necessary (estimated requirement is 1200 lb hoop strength, 600 lb or less axial strength). Its puncture resistance was approximately equal to the target value of 200 lb. The values for the 8 layer structure were somewhat lower than the target values, except for the axial strength. Hoop strength was higher, however, than that of the currently used drum material which we measured to be 950 lb/in.

In spite of these results, we recommended proceeding with the 8 layer braided structure. We did not have a large enough cast sample to determine strength uniformity, but the structure is such that one would expect variability in 1 inch wide specimens to be high. Thus, the average of 5 or 10

determinations would almost certainly be different from 1140 lb/in, which was the result obtained from only one specimen. Indeed, the ratio of the strength values for the 10 layer and 8 layer specimens should be about 10/8 = 1.25, rather than the ratio obtained 1620/1140 = 1.42. This was a good indication that 1140 lb/in was a low value for the 8 layer structure, and perhaps 1620 lb/in was a high value for the 10 layer structure. It is also true that the value obtained for puncture resistance is very dependent on the angle of the penetrating blade relative to the yarns in the structure. Our past experience led us to believe that the 158 lb was a low value, and that the average of many measurements would be closer to 200 lb.

Perhaps the most important reason for recommending the 8 layer structure was that it provided maximum flexibility and reasonable properties at the lowest cost. Failing any actual experience with drums made in this way, we believe the choice of the 8 layer braid, 1 layer knit structure to be the best decision for manufacturing the first two test drums.

Coating Technology

Since one of the prime objectives of the present work was to develop an elastomeric coating system that would remain flexible at -60°F, be readily available, and be processable using standard injection molding technology, only a limited number of elastomeric polymer systems could be considered.

Five elastomer types which were considered initially were polyurethanes, polysulfides, fluorophosphazines, fluorosilicones and fluorocarbons. However, all of these except polyurethanes proved to be unacceptable because they could only be used in solvent systems, which cannot be used in injection molding. Polyurethanes, which are available as 100% solids systems, were chosen as the most promising polymer type to be investigated. All of our work was concentrated, therefore, on the development of a polyurethane system suitable for injection molding, and having the desired fuel and water resistance coupled with low temperature flexibility.

The investigation of a suitable polyurethane initially indicated that a blend of a polycaprolactone (for fuel resistance) and polyether (for low temperature flexibility) urethane might well have desirable properties. The first in this series was based on an Upjohn polycaprolactone:

Formulation 2642-11-2	
Prepolymer Upjohn 2102-80-AE	100
Benzoflex T-150 plasticizer	10
PTEMG (1000) polyether glycol	29
1,4 Butanediol	6

A second formulation was based on an Essex polycaprolactone blended with a polyether:

Formulation 2642-16-1	
Prepolymer Essex Betathane E/23-800	100
Benzoflex 9-88 plasticizer	10
PTMEG (1000) polyether glycol	28.4
1,4 Butanediol	5.96

A slight modification of this formulation consisted of a pre-reacted blend of polycaprolactone and a polyether.

Formulation 2642-24-2	
Essex Bethane U855.06	100
Benzoflex 9-88	10
PTMEG polyether glycol	26.83
1,4 butanediol	6.04
NPDA (N-phenyl diethanolamine)	0.96

Other urethanes evaluated at this time were:

Upjohn Castethane CPR2141 polycaprolactone
Upjohn Castethane CPR2148 polypropylene glycol
Upjohn Castethane CPR NS285-91-1 polycaprolactone blend
Essex Betathane E23-700 blended polyether
Adiprene L-100 polyether.

All of these compounds became brittle at -60°F except Adiprene L-100. However, its fuel diffusion rate was very high (greater than 1) and solvent swell was excessive. All of these compounds were discarded as being unsuitable.

In addition, a series of formulations was made up using polyether urethane prepolymers for low temperature flexibility:

	2642-25-1	2642-25-2	2642-25-3	2642-25-4
Prepolymer type	Essex Betathane E23-701	Essex Betathane E23-710	Upjohn Castethane CPR-2162	Du Pont Adiprene M-483
Prepolymer parts	100	100	100	100
Benzoflex 9-88	10	10	10	10
Curative Blend				
PTMEG	27.96	27.27	34.52	22.09
BDO	5.57	5.43	6.88	4.40
NPDA	1.01	0.99	1.25	0.80

The results of tests made on test strips of each of these formulations are given in Table 4. All characteristics of the caprolactone samples (11-2, 16-1, 24-2) were satisfactory except low temperature stiffness. They were discarded because of an excessive modulus at -60°F. This was true also for 25-1, a polyether formulation, which was discarded because of low temperature embrittlement. Formulations 25-2, 3 and 4 all seemed potentially suitable, and the compound based on Adiprene M-483 was selected as being the most promising. Its major deficiency was a 60% volume swell in test fuel B.

Table 4: Drum Coating Characteristics

Formulation 2642-	11-2	16-1	24-2	25-1	25-2	25-3	25-4	25-5
Manufacturer	Upjohn	Essex	Essex	Essex	Essex	Upjohn	DuPont	DuPont
Urethane Type	Caprolactone	Caprolactone	Lactone/Ether	Polyether	Polyether	Polyether	Polyether	Polyether
Name	Castethane CPR2102-80	Betathane E/23-800	Betathane E/U855-06	Betathane E/23-701	Betathane E/23-701	Castethane CPR2162	Adiprene M483	Adiprene M483
Hardness, Shore A	65	74	68	74	76	65	70	74
G at -60°F, psi	93,000	28,000	53,000	Fractures	4,100	1,780	820/955	810
Strength, psi	7,060	1,990	2,270	---	2,570	1,640	2,000/2,400	2,900
Elongation, %	520	670	540	---	570	630	720/540	515
Strength Retention in Fuel B, %	80	82	75	---	50	73	69/58	45
Volume Swell, %	17	20	38	---	53/60	54/60	69/58	61
Tear Strength, lbs/in	523	231	249	---	190	---	175	190
Fuel Diffusion Index	0.35	0.3	0.45	---	---	0.9	0.5*	---
Water Resistance	Good	Good	Excellent	Excellent	Excellent	Excellent	Excellent	Excellent

*After one week exposure, fuel diffusion index continued to drop slowly. After one month the index was less than 0.3 and still dropping.

Formulation 2642-25-5 was made in an attempt to reduce volume swell by reducing the plasticizer content. The following shows the formulation change.

	2642-25-4	2642-25-5
Prepolymer adiprene M-483	100	100
Benzoflex 9-88	10	0
PTMEG polyether glycol	22.1 (29 mole %)	19.05 (25 mole %)
BDO (1,4 butanediol)	4.4 (64.2 mole %)	4.7 (68.2 mole %)
NPDA (N-phenyl diethanolamine)	0.8 (5.8 mole %)	0.8 (5.8 mole %)

The volume swell did not improve and loss in strength after fuel immersion increased. At this point, the plasticized formulation, 2642-25-4, was considered as having a reasonable balance of properties, and was recommended for use in the production of a LIM process drum sample.

In the LIM process (liquid-injection-molding) it is important to control both the gel time and surface wetting properties of the polymer system. Dabco tri-functional amine catalyst and "Multiflow" wetting agent are added at the 0.05% and 0.1% levels to control these properties. A gel time of 10-15 minutes was achieved with good wetout of the molding surfaces.

For the proposed LIM manufacturing process, the core or mandrel must be coated or covered with a liner having essentially the same properties as the drum system. This inner liner will be applied by spray application and will require ten passes or applications to obtain the necessary thickness.

The following spray formulation, based on 2642-25-4 with a solvent carrier, was used in preliminary trials:

Liner Spray Formulation

Part A - Adiprene M-483	100
Benzoflex 9-88SG	10
Multiflow	0.1
Solvent*	73.4

*Solvent (50/50 by weight M-xylene/toluene).

Part B - 1,4-butanediol	4.4
PTMEG (Teracol 1000)	22.09
NPDA	0.8

When preparing Part A, the Adiprene with Benzoflex and Multiflow were preheated to 170°F and then the solvent was added. Part B was preheated to 100°F and then added to Part A. The resultant mixture was then degassed. Spray-coating the prototype inner liner was accomplished utilizing a pressurized tank-spray gun system. A minimum of 45 minutes was allowed between passes for proper gelling. It was also advisable to heat the mandrel with liner coating to 100-140°F between passes to insure complete removal of the carrier solvent.

Upon completion of the liner preparation and curing, the Kevlar reinforcing will be applied to the mandrel and then the mandrel will be placed and locked into the drum mold. The LIM process operation will be accomplished using the 2642-25-4 formulation with the following modifications:

Part A - maintain and process at 170°F	
Adiprene M-483	100
Benzoflex 9-88SG	10
Multiflow	0.1
Part B - maintain and process at 80°F	
1,4-butanediol	16.41
PTMEG-Teracol 1000	80.62
NPDA	2.97
Dabco Amine Catalyst	0.05
Color	2

Injection ratio Part A/Part B = 100/24.7 = 4.05.

Bonding to Metal Fittings

The drum hardware is to be installed in holes in either end of the carcass, in which two metal rings which are bolted together through the drum carcass are to be bonded to the coated fabric with an adhesive system. The hardware was required to be sulfuric acid anodized per MIL-A-8625, Type II, Class 2. We experienced great difficulty in obtaining significant adhesion to such a surface, and found that none of the adhesive suppliers could recommend an adhesive system for a sulfuric acid anodized aluminum surface. This was confirmed by chemists at MERADCOM who said that good bonding could only be achieved by using phosphoric acid anodizing.

The hardware which we were to use was to be removed from used fuel drums. There was no way of knowing what type of finish this hardware had received. However, since it had been used in fuel drums, one had to assume that it was satisfactory.

Tests in the laboratory were done using sulfuric acid anodized aluminum strips. In order to achieve any reasonable adhesion, it was necessary to remove most of this anodizing by sanding the surface and then cleaning with a solvent. This procedure was also followed when installing the actual hardware, which had to be thoroughly cleaned mechanically and chemically to remove all traces of any adhesive which had been used in the fabrication of the drums from which it was removed.

Five adhesive systems were tested, with the following results (see Table 5).

The adhesive system which was finally adopted was as follows:

1. Clean the anodized surface by mechanical sanding followed by solvent cleaning.

2. Apply a prime coat of Hughson Chemical's metal primer #205.

3. Apply and dry a coat of Chemlok 233.

Table 5: Bonding System Characteristics

Supplier	Identification	Type	Adhesion (lb/inch) Original	Adhesion (lb/inch) After Fuel B Aging
Hughson Chemical Co.	Versilok 204/accelerator #4	acrylic	70	10
Hughson Chemical Co.	Tycel 7002/7202	2-part urethane	19	3
Hughson Chemical Co.	Chemlok 233/metal primer 205 with Adiprene L-100	organic polymers with isocyanate	25	17
Hughson Chemical Co.	Chemlok 233/metal primer 205 with Adiprene M-483	--	70	25
Bostik Division	Bostik 7376/Boscodur #4	--	30	10

4. Apply a fresh coat of Adiprene M-483 (formulation #2642-25-4) to the coated fabric surface.

5. Clamp and heat to 250°F for 30 minutes.

This gave excellent initial peel strength. The relatively poor resistance to fuel exhibited by the test strips was attributed to the presence of the sulfuric acid anodizing. If the hardware which was used had a different finish, the resistance might well have been improved. There was no way of checking this without destructively testing the finished drum.

Phase II: Drum Fabrication

As stated earlier, the general purpose of this work was twofold:

(a) To develop a coating material suitable for drum manufacture which will have the required fuel resistance and will remain flexible to -60°F.

(b) To demonstrate the feasibility of a novel procedure for manufacturing such drums by producing and delivering two drums for tests to be conducted in the Arctic.

The coating material, based on Adiprene M-483, was developed in Phase I of the work. The basis for its choice was described in the Phase I section of this report.

Because only two drums were required to be fabricated in Phase II, all manufacturing steps were designed to be as simple and inexpensive as possible. The procedure requires two pieces of precision tooling: (1) a mandrel on which the drum will be built, having dimensions accurate to ±0.05"; (2) a mold to enclose the mandrel, urethane liner and fiber reinforcement to permit injection molding of the urethane cover to a total thickness of approximately 0.25". Because clearances between the mandrel and the mold are so small, the mold dimensions also had to be controlled to ±0.05". After molding, the mandrel had to be capable of being removed through 10-1/2" diameter holes on either end of the drum.

In order to keep the tooling as simple as possible, we initially planned to use a mandrel built out of a plaster/fiberglass mix. Unfortunately, our first attempts to build a drum on such a mandrel failed because the mandrel would not withstand the stresses imposed in the molding operation. Accordingly, a mandrel made from a fiberglass/polyester lay-up was designed which could be dismantled inside the fabricated drum and removed through the end holes. Using this mandrel, two drums were made, tested and delivered.

Final Mandrel Design

The fuel drum is fabricated around an inner rigid mandrel, which establishes the size and shape of the finished drum. The mandrel is collapsible so that it can be disassembled and removed through the end openings of the drum when fabrication is completed. For this program, with the object to produce

only two fuel drums, the mandrel was designed as an item of temporary tooling, and was constructed from glass reinforced polyester resin. Although this construction is suitable for several repeated uses, a more permanent metal mandrel would be necessary for the manufacture of fuel drums in production quantity lots.

The mandrel design consists of a central shaft surrounded by a ring of outer members similar to barrel staves, or the outer surface of orange segments (see Figure 15). The pointed ends of the staves were bolted to flanges positioned on the central shaft. These flanges had a large hub diameter which generated the circular holes in the ends of the fuel drum (ref. MIL-D-23119D). Projecting pins on the ends of the staves provided a circular array positioned to generate the ring of bolt holes for the hardware at each end of the fuel drum.

Special design features were required to permit the disassembly, manipulation and removal of the mandrel staves through the small openings in the fuel drum. These included one master stave, with parallel surface edges next to the two adjacent staves, to permit movement of this stave in a radially inward direction after disconnecting from the flanged central shaft. In addition, all of the staves were made in two pieces, with the curved ski-tip position at one end being removeable from the rest of the stave (see Figure 16). This permitted the collapse of the staves inwardly and also simplified the positioning and maneuvering of each stave through the small diameter access opening. The bolted connection of the removeable stave ends was made up with specially fabricated long stem bolts, with a wrenching hex head positioned for accessibility through the hole in the drum.

Assembly of the mandrel is a straightforward operation, since all of the parts are exposed and accessible. Briefly, the flanges are positioned on the central shaft, which is then supported horizontally on a cradle with pedestals at both ends (this shaft is a hollow tube, longer than the fuel drum, with extensions at both ends). The two pieces of each stave are joined together, and the completed staves are positioned around the shaft one at a time, and bolted to the shaft flanges (see Figure 17). The assembled mandrel is then liberally coated with a release agent, and the seams between the segments covered with masking tape (see Figure 18). It is now ready for the drum manufacturing process which is described in detail elsewhere in this report.

After the fuel drum has been fabricated and finish cured, the mandrel is removed from inside the finished fuel drum. The bolts holding the ends of the staves to the shaft flanges are exposed on the outside, and can be removed. The flange at one end of the shaft can then be slid off over the end of the shaft, and the shaft itself with the other flange can be taken out of the opposite end of the drum. Now the openings in the ends of the drum are exposed, but the mandrel staves are supported in place by the surrounding drum fabric. The master drum segment can next be separated into two pieces by releasing the long stemmed bolt, and can then be maneuvered out of the drum access port. Disassembly and removal of all of the remaining stave segments is now possible, although care must be taken to prevent tumbling of the staves and possible damage to the inside of the drum while working through the restricted openings.

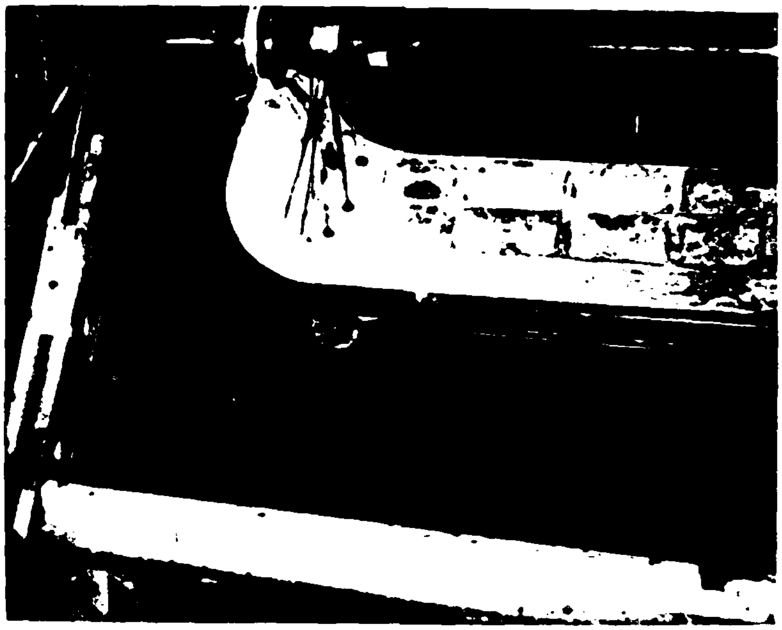


Figure 15. Segmented Mandrel Design

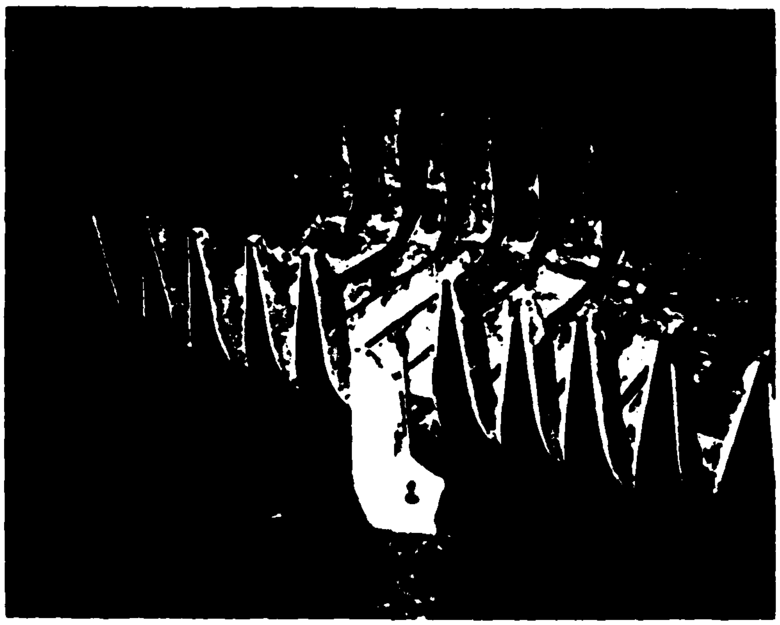


Figure 16. Mandrel Stave Design

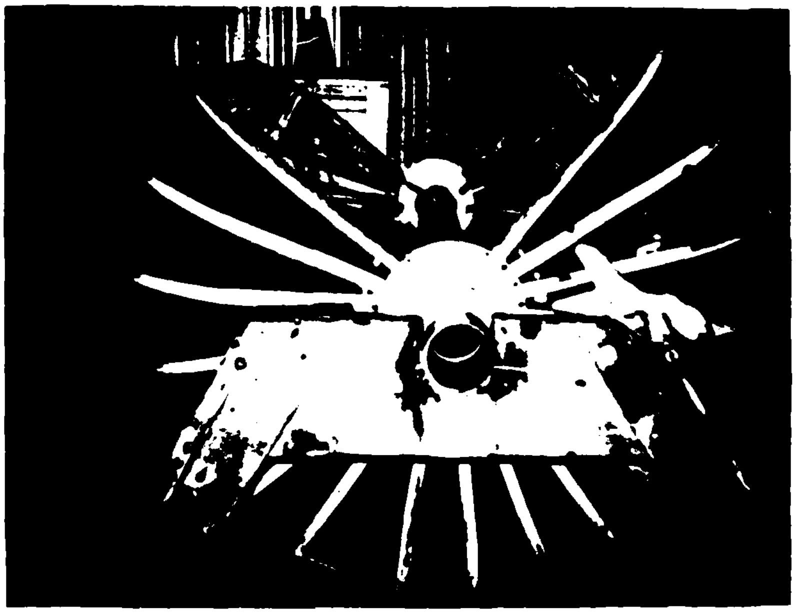


Figure 17. Mandrel Assembly

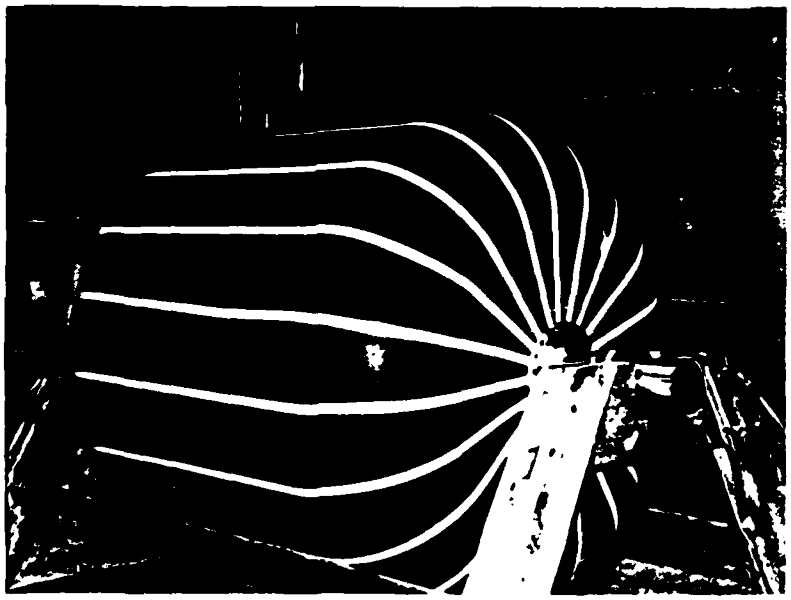


Figure 18. Mandrel Ready for Application of Urethane Liner Coat

Outer Mold

A fiberglass/polyester outer mold was made by laying up sections on a built-up form made to simulate the desired outer dimensions of the finished drums. The inside dimensions of the assembled outer mold were such as to give 1/4" clearance from the mandrel all around the body, tapering at each end from 1/4" at the shoulder to 1/2" at the hub. The mold was made in six pieces, consisting of four end segments and two semi-cylindrical body segments. Each of these was mounted on the central shaft to ensure concentricity with the inner mandrel. Assembly around the coated and fiberglass-reinforced mandrel was effected by bolting sets of 3 together to form two halves, and then mounting these halves around the covered mandrel on the central shaft (see Figure 19). The mold seams were made leak-proof by using an internal sealing compound, as described later.

The mold was supplied with an inlet port at one end and an outlet port at the other. In the molding operation the central shaft was supported in a vertical position so that the urethane compound was pumped into the inlet port at the bottom and air and, eventually, compound escaped through the outlet port at the top.

Drum Manufacture

The mandrel was coated with Adiprene M-483 by spraying the first coat and brushing an additional coat to obtain a thickness of approximately 0.1". After partial curing in an oven at 190^oF, the body and two ends were covered with a Kevlar ballistic fabric of the following construction (see Figure 20):

plain weave 17 x 17	1500 denier Kevlar
7.0 oz/yd^2	750 x 750 lb/inch

The body was then covered with 8 layers of triaxial braid which extended about halfway around each of the shoulders, and overlapped by about 6" the fabric pieces which covered the ends of the drum (see Figure 21).

Finally, the assembly was enclosed in the mold and the cover coat pumped in from a liquid injection molding machine which blended the two heated components in the desired proportions and delivered the mix at a pumping pressure of a few psi. Approximately 250 lb of urethane was used. The compound was adjusted to have a gel time of about 30 minutes. The mold was filled in 27 minutes. The drum was cured in an oven at 250^oF overnight, and then the oven was allowed to cool slowly.

The mold was removed to reveal a good cover except for the following flaws:

(a) An air bubble which had been trapped in the top of the mold prevented the urethane from coating an area approximately 6" x 12".

(b) Zinc chromate paste, which was used to seal the mold joints, was extruded into the interior of the mold when the joints were tightened. This formed a groove in the molded urethane drum cover along the joint lines.

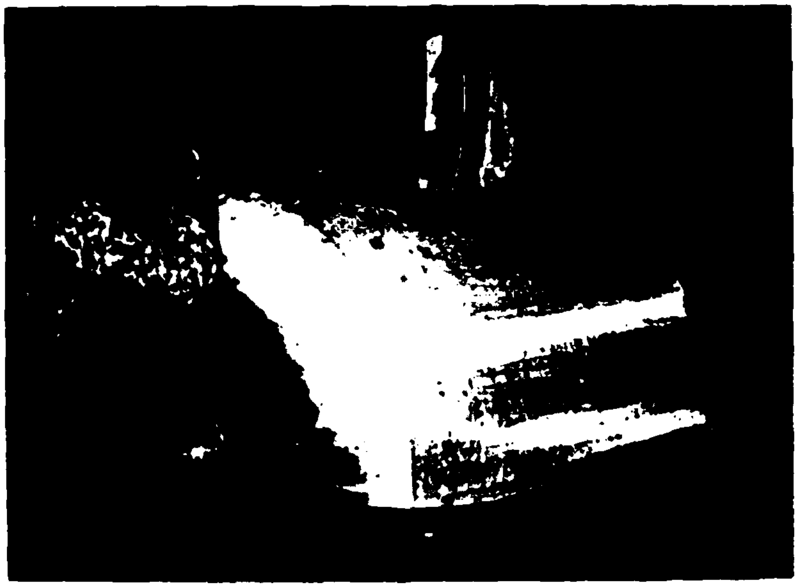


Figure 19. Mold Assembly Around Covered Mandrel

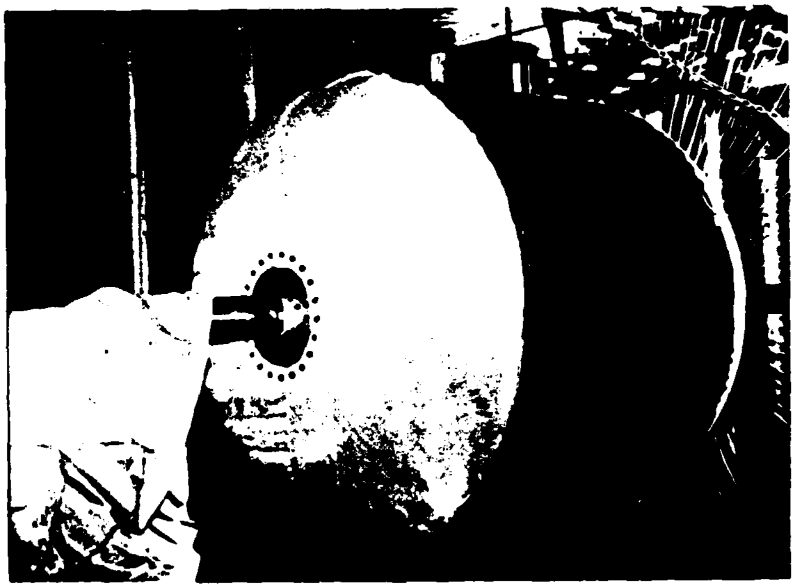


Figure 20. Fabric Covering Ends of Coated Mandrel

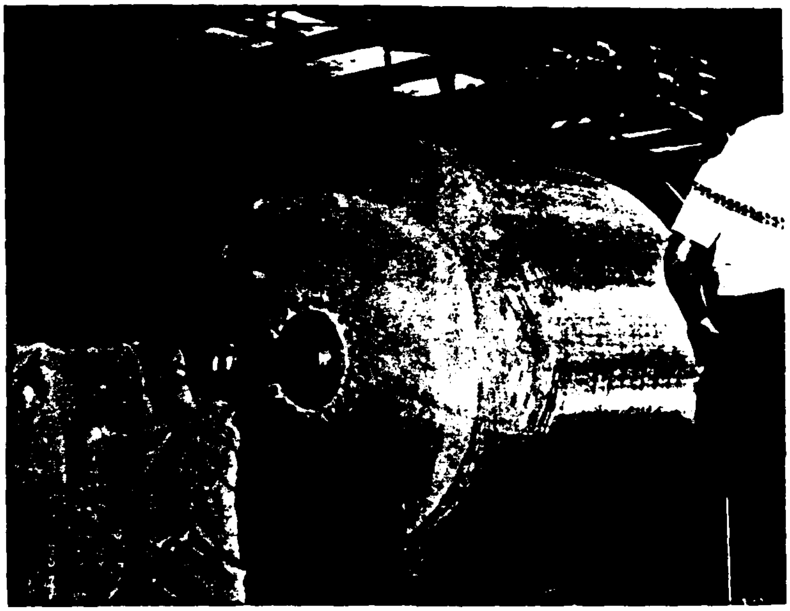


Figure 21. Braided Cover Being Added to Body of Drum

(c) Due to a lack of perfect concentricity, the coating on one small area along one of the drum shoulders was thinner than it should be.

(d) When the two halves of the mold were being closed, a slight cocking of one half relative to the other required filling to get a tight joint.

None of these faults was serious, and M-483 urethane was applied to cover them, although this gave the surface a patched appearance. Functionality should not be affected. Changes in the molding procedure for the second drum were made in an attempt to overcome these problems.

The hardware was installed by using the Chemlok/Adiprene M-483 adhesive system developed in Phase I. Because of the tendency of urethane to flow under pressure, it was necessary to tighten the bolts which attach the hardware to the drum several times over a period of 2-3 days. It would also be advisable to retighten all of these bolts when the drum is acclimated to Arctic conditions.

Inflating to 6 psi with air revealed no leaks (see Figure 22). The drum was then filled with water and the pressure gradually increased. At 15 psi some sounds indicated that some yarn breakage was occurring. We believe that this was the sewing thread in the seam which attached the two fabric strips together to form the end panels. The pressure was not increased over 15 psi, and we recommend that operating pressures do not exceed 10 psi for this drum. No leaks were observed.

Because of the observations made during pressure testing of the first drum, the end reinforcement was improved in the second drum. Two pieces of the fabric used for reinforcement were needed to obtain sufficient width to cover the end of the mandrel. It was believed that the seam used to attach these two pieces was inadequate in the first drum. Consequently, a stronger seam was used on the second drum, by using a 1-1/2" overlap of the selvage edges, and a diamond pattern of seaming, similar to that used in parachutes to seam webbing. The seaming thread was Kevlar, Size E. In order to reinforce the ends even more, two layers of fabric were used on each end, in place of the single layer used in the first drum. One was applied to the end directly over the coated mandrel, and the second after 4 layers of braid had been added.

A tie-coat, consisting of a 3% solution in MEK of 1 part of silane and 5 parts of isocyanate, was sprayed onto the fabric and braid reinforcement to promote adhesion to the urethane.

The seams of the mold were sealed with a strip of cured silicone rubber in place of the zinc chromate paste. This gave an excellent seal and eliminated the problems caused by extrusion of the paste from the mold seams, causing marks in the surface of the first drum which had to be scraped and filled.

In an attempt to improve air escape from the mold during the molding operation, 4 small vents were added to the top of the mold to augment the one large vent used in molding the first drum. Also, by means of a very slight decrease in the amount of catalyst used, the gel time of the urethane formulation was increased from 30 minutes to approximately 40 minutes. To reduce

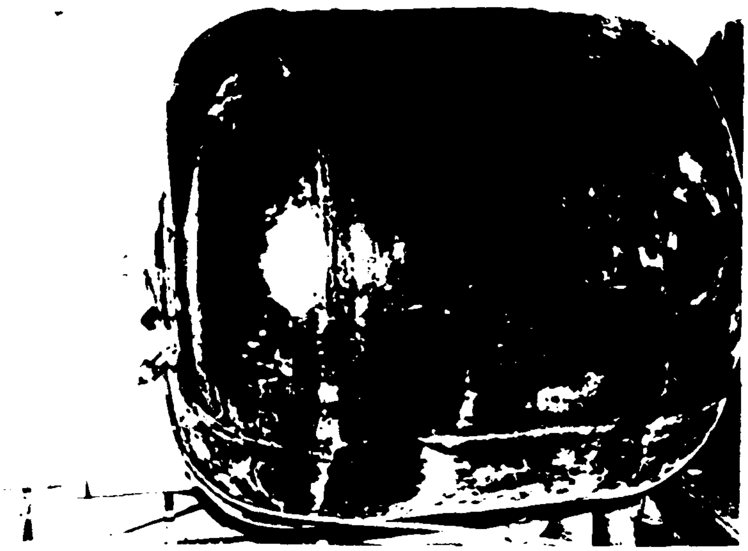


Figure 22. Drum Undergoing Pressure Test

the viscosity of the molding compound, the temperature of the mold was increased from 150°F to 170°F. This reduced the time required to fill the mold from about 27 minutes to 21 minutes.

All of these changes resulted in an improved molding operation, and a better final product. There were still some flaws, however, requiring patching. Some air bubbles still became trapped around the upper shoulder. Although these were not as large or as numerous as in the first drum, their presence indicates that improvements still need to be made in the molding operation. Also, as in the first drum, there was an area on the top end of the drum where penetration through the fabric reinforcement had not been sufficient to give good adhesion to the urethane liner. As before, this was patched by peeling back the inner liner, spreading additional urethane onto and through the exposed fabric, and resealing the liner flap. The hardware was installed by the same technique used for the first drum. Again periodic retightening of the bolts was needed to accommodate compound creep.

Pressure testing of this drum was more satisfactory than in the case of the first drum. There were no indications, either audible or visual, that indicated weak spots in the drum. Again, however, the test was taken only to a water pressure of 15 psi, since we were still dealing with a drum containing minor flaws, and we did not want to risk catastrophic failure of this prototype product. We believe that 15 psi is significantly higher than any pressures which will be encountered in the proposed use tests in the Arctic.

Although no more drums are to be made under the existing contract, certain changes in the manufacturing procedure would be made if and when additional drums are required. They are:

(1) A different fabric construction would be used for the reinforcing woven fabric. We would look for a basket weave made from 2 ply, 1500 denier Kevlar, woven about 17 x 17 at a width of not less than 72"; or, even better, a basket weave woven from 3 ply, 1500 denier Kevlar, woven about 17 x 17 at a width of not less than 72"; or, better still, a basket weave woven from 3 ply, 1500 denier at about 12 x 12. This would be strong enough and large enough to cover the ends in a single unseamed layer, and have large enough pores to ensure easy and adequate penetration of the molding compound.

(2) Additional care must be taken to minimize entrapment of air bubbles at the upper shoulder. This will be helped by using a more open fabric structure, but could also be aided by saturating the fabric prior to insertion in the mold by a spray coat of urethane, by rocking the mold, or by other techniques which may come to mind.

We are satisfied that we have demonstrated that the technique is a viable one, and that the remaining problems are minor and capable of being solved. We believe the drums which were produced were structurally sound (the second better than the first) and that the flaws which existed were functionally unimportant.

Arctic Test Results

Exposure of the two prototype drums in the Alaskan winter revealed a serious fault which had been undetected in the laboratory testing which had been done. The material became excessively stiff, so that a man could stand on an empty drum without it collapsing.

Sample slabs of the urethane molding compound had been cast at the time the drums were molded. These were sent to the Belvoir R&D Center for evaluation in the way that the compound had originally been tested. At that time, after 24 hours at -60°F, the coating had a stiffness modulus of 900 psi, which was well below the target maximum of 10,000 psi, and lower than any other acceptable compound tested. It was on the basis of this evaluation that M-483 had been approved as the most promising compound for drum manufacture.

Retesting of the compound used to make the two drums gave the following results:

Exposure Time at -60°F	Condition	Stiffness Ratio Drum 1	Stiffness Ratio Drum 2	Stiffness Moduli (psi) Drum 1	Stiffness Moduli (psi) Drum 2
2 days	unextracted	5.5	5.6	1160	1030
	extracted	6.7	6.9	1560	1400
7 days	unextracted	25.7	17.4	5420	3200
	extracted	43.4	31.4	10260	6420

The results after 2 days exposure at -60°F show reasonable agreement between the two drums, as well as with the value given above which was obtained during Phase I of this work. The results after 7 days show clearly that the stiffening which was seen in the Arctic is slow to develop, but of sufficient magnitude to explain the behavior of the drums. It is possible that the modulus values would rise to even higher values for exposures longer than 7 days.

The problem appears to be a slow, progressive crystallization of the urethane when exposed to low temperatures. This is apparently the result of the particular chemical structure of Adiprene M-483. Usually, however, crystallization is not hard to inhibit by the presence of a foreign substance. It was thought that improvement in the low temperature properties might result from the blending in of small quantities of another polyurethane. A possible candidate might be, for example, Upjohn's Castethane CPR 2141 polycaprolactone, which had a stiffness modulus at -60°F of only 1000 psi.

Small test slabs were prepared in which 5% or 10% of Castethane was blended into M-483 resin. Tests by MERADCOM indicated that the low temperature flexibility had been improved, but not enough to be useful. However, this was only a simple trial to indicate the feasibility of a concept, not to develop an optimum blend.

The possibility of inhibiting the low temperature crystallization of M-483 by disrupting the symmetry of the structure has been demonstrated. A more attractive approach was also examined, in which a small amount of a low molecular weight polyol was blended into the regular polyol component of the

urethane. This was sufficient to disrupt the chain symmetry so that crystallization could not easily occur. A simple subjective evaluation of stiffness indicated that this was effective for exposures at -20°F for two weeks. A sample was forwarded to Belvoir R&D Center for their evaluation. The results of their measurements are given below.

	-40°F		-60°F
Exposure Time	Stiffness Ratio	Stiffness Modulus (psi)	Stiffness Modulus (psi)
1	93	7570	9130
3	131	10260	12530
7	169	12530	15520

These values can be compared to the values given on page 33 for the unmodified compound which was tested at -60°F. The modification has resulted in a higher initial modulus, but the rate at which crystallization is occurring seems to have been slowed down. Even more encouraging is the relatively small increase in stiffness which occurred in the modified compound between -40° and -60°F. This is a temperature range where large increases in stiffness commonly occur.

Clearly this single trial has not solved the problem of low temperature stiffening. However, the results are encouraging enough to indicate that a study of the structural charges brought about by this method of modification would be warranted, with a view to establishing whether an appropriate additive can provide the improved low temperature flexibility that is needed. This study is beyond the scope of the present contract.

Conclusion

The feasibility of a totally new method of making fuel drums has been demonstrated. Although some manufacturing problems still exist, relatively simple means of solving them are available. Unfortunately, a totally suitable compound for use in the Arctic has not been found, but the possibility of modifying the chemistry of the compound to improve its low temperature flexibiity has been demonstrated. A study of limited scope to explore the practicality of this approach was proposed but has not been funded at the present time.

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